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TC 1700

PATENT
Attorney Docket No. 5725.0429-00
Customer No. 22,852

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Marie-Pascale AUDOUSSET

Application No. 09/335,742

Originally filed: June 18, 1999

CPA Filed: April 10, 2001

For: DYE COMPOSITION CONTAINING
1,8-BIS(2,5-DIAMINOPHENOXY)-3,6-
DIOXA OCTANE, AN ADDITIONAL
OXIDATION BASE AND A COUPLER
AND DYEING PROCESS (As Amended)

Group Art Unit: 1751

Examiner: C. Liott

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

DECLARATION UNDER 37 C.F.R. 1.132

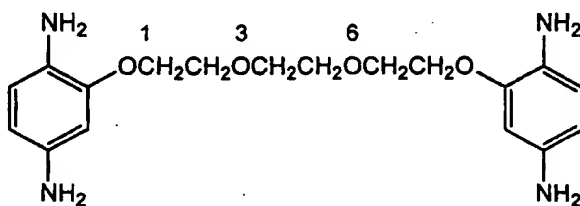
I, Marie-Pascale AUDOUSSET, declare and state that:

1. I am a French citizen, residing at 1 Allée Louis Jouvét, 92600 Asnières, France.
2. I have been awarded a degree in Chemical Engineering from the École Nationale Supérieure de Chimie de Paris, and am a Doctor in Organic Chemistry.
3. I have been employed by L'ORÉAL since 1986 and I am presently a Laboratory Supervisor of the hair dyeing research laboratory at L'ORÉAL. During my employment at L'ORÉAL, I have been engaged in research and development regarding hair dyeing.
4. I am the inventor of the invention set forth in the above-identified application.

5. Given my education and experience, particularly in the area of hair dyeing, I consider myself able to provide the following testimony as to the proper structure and nomenclature of 1,8-bis-(2,5-diaminophenoxy)-3,6-dioxaoctane.
6. Those of ordinary skill in the art would readily recognize that the chemical name 1,8-bis-(2,5-diaminophenoxy)-3,5-dioxaoctane used in the present application contains a typographical error. The presently claimed first oxidation base should instead be designated 1,8-bis-(2,5-diaminophenoxy)-3,6-dioxaoctane.
7. In the present application, it is stated on p. 2 that it was known, *e.g.*, in PCT application WO 92/13824, to use 2,5-diaminophenoxyoxaalkanes as oxidation bases, optionally in combination with couplers. It is then stated (in the paragraph bridging pp. 2-3) that the present inventor (myself) had now discovered, surprisingly and unexpectedly, that the combination of 1,8-bis-(2,5-diaminophenoxy)-3,5-dioxaoctane with at least one second suitably selected oxidation base and at least one coupler gave intense colorations with improved properties.
8. As discussed below, my laboratory notebooks from November 18, 1997, to December 1, 1997, show that indeed the WO 92/13824 compound was the one on which I based this invention. Although the compound is referred to as "...3,5-dioxaoctane" in example 1.1 of the WO document, it is clear from the structural formula (I) at pages 2, 3, and 12 of the WO document that this compound should properly have been designated "...3,6-dioxaoctane."

9. In the WO document, the compound of formula (I) contains a chain having repeating ethylene oxide units, $-(OC_nH_{2n})_x-$, where n is 2 or 3 and x ranges from 1-3, and linking two 2,5-diaminophenoxy units. Thus, in the ethylene oxide chain [specifically $-OC_nH_{2n}(OC_nH_{2n})_xO-$ when including the phenoxy oxygens in the "chain"] linking the 2,5-diaminophenoxy units, there must be at least two carbon atoms between each occurring oxygen atom. Such a compound is a "...-3,6-dioxaoctane."

10. This nomenclature is derived as follows: starting from the left-most carbon atom of the chain, the positions of the non-phenoxy oxygen atoms are counted and labeled. Thus, where $n=2$ and $x=2$, as in example 1.1 of the WO document (p. 7), the chain of ethylene oxide units yields non-phenoxy oxygen atoms only at the 3 and 6 positions.



11. Thus, in view of the above, the present application should recite "1,8-bis-(2,5-diaminophenoxy)-3,6-dioxaoctane" instead of "...-3,5-dioxaoctane" throughout the specification and claims. One skilled in the art would recognize this error and would know how to correct it.

12. I have also attached copies of my laboratory notebook pages dated from November 18, 1997 to December 1, 1997, dated before the filing date of the present application and before the filing

date of French priority application FR 98 07793, filed June 19, 1998. Pages 168-171 of notebook No. 969 document my first description of the claimed composition.

13. Notebook page 168, dated November 18, 1997, depicts the 3,6-compound. The 3,6-compound is named under the internal code "A" which has the salt form $4\text{HCl} \cdot \text{H}_2\text{O}$.

14. Notebook page 169, dated November 18, 1997, discusses WO 92/13824 as relevant prior art in that it is the base patent for the 3,6 compound.

15. Notebook page 170, dated December 1, 1997, lists two examples of hair dyeing compositions including A (the 3,6-compound), a second base and a coupler.

16. Notebook page 171, dated December 1, 1997, provides a table showing test results of hair dyed with a composition containing A, the 3,6-compound.

17. These notebook pages show that at all times I contemplated a 3,6-compound for use in a hair dyeing composition before the filing date of the present application and the priority application.

18. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: January 10th, 2002

By: Marie-Pascale Audousset
Marie-Pascale Audousset

168

CAHIER N° 929

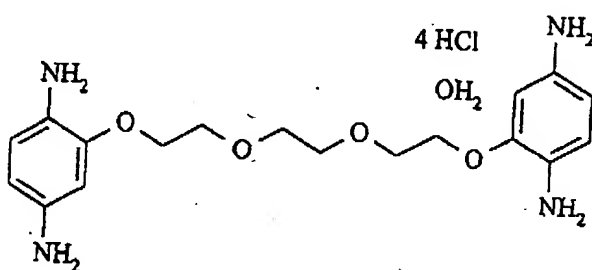
Suite de la Page

Confidentiel

18.11.81

M. Audouin

Colipa A + 1 base sélectionnée + 1 coupleur
sélectionné
Projet de brevet - CL97/078

Structure AProblème posé :

L'utilisation de la base d'oxydation A est connue en coloration d'oxydation. Les résultats obtenus sont cependant peu puissants.

Solution apportée :

Nous avons montré de manière surprenante que l'association de ce colorant à une base d'oxydation convenablement choisie et à un coupleur

SIGNATURE

M. Audouin

Suite page 169

NOMBRE DE LIGNES UTILISÉES

2 à 28

DATE

18.11.81

TRANSMIS A ET COMPRIS PAR (Noms et signatures)

A. de Labbey

DATE

01/12/81

TEMOIN (Noms et signatures)

H. LAKHOUSAY

DATE

1.12.81

CAHIER N° 979

169

Suite de la Page 168

convenablement choisi permet d'obtenir des puissances de coloration nettement supérieures.

Art antérieur :

Le document qui nous ont semblé pertinent sont les suivants :

Henkel WO92/13824 Pr 31/1/1997¹ qui est le brevet de base¹ de la molécule A.

Nous avons fait faire un recherche complémentaire par CAS des brevets Henkel postérieurs au brevet de base et citant le colorant Colipa A. Six autres brevets sont ressortis, ils ne concernent que les réactions colorées et le A est cité parmi les amines pouvant réagir avec les sites cétoniques. Ces documents ne sont donc pas gênants.

Description de notre invention :

La composition colorante est constituée du colorant A, associé à une autre base et au moins un coupleur.

Bases d'oxydation : pas de limitation particulière, ppd et dérivés, pap et dérivés, oap et dérivés, bases hétérocycliques de type diaminopyrazole ou pyrazolopyrimidine.

Coupleurs d'oxydation : pas de limitation, MAP et dérivés, MPD et dérivés, résorcine et dérivés, coupleurs hétérocycliques.

Les colorants sont aux concentrations habituellement décrites.

Le pH de coloration n'est pas limité.

L'agent oxydant est l'eau oxygéné ou autres.



Suite page 170

SIGNATURE <i>Ngidouner</i>		NOMBRE DE LIGNES UTILISÉES 2 à 30	DATE 18.11.97
TRANSMIS A ET COMPRIS PAR (nom et signature) <i>A. de la Bette</i>		DATE 01/12/97	TEMOIN (Nom et signature) <i>H. CAUHOUSY</i>
			DATE 1.12.97

CAHIER N° 979

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A + 1 base + 1 coupleur illustrations

Vous trouverez ci-joint les illustrations du projet CL97/078.

COLORATION D'OXYDATION - Support simplifié :

pH basique

composition colorante

A	0.75 10-3 mol%
autre base	1.5 10-3 mol%
coupleur	3 10-3 mol%
alcool éthylique à 96°	18 g
Métabisulfite de sodium en solution aqueuse à 35%	0.68 g
sel pentasodique de l'acide diethylene	1.1g
tri amino pentacétique	10g
ammoniaque à 20%	qs 100 g
eau déminéralisée	

pH neutre

A	0.75 10-3 mol%
autre base	1.5 10-3 mol%
coupleur	3 10-3 mol%
alcool éthylique à 96°	18 g
Métabisulfite de sodium en solution aqueuse à 35%	0.68 g
sel pentasodique de l'acide diethylene	1.1g
tri amino pentacétique	10g
K2HPO4/KH2PO4 (1.5M/1M)	qs 100 g
eau déminéralisée	

Mode d'application :

La composition colorante est mélangée p/p avec H₂O₂ 20 V.
Le mélange ainsi réalisé est appliqué sur des cheveux gris à 90% de blancs, permanents
raison de 30 g pour 3 g de cheveux, pendant 30 mn.
Les cheveux sont ensuite rincés, lavés avec un shampoing standard et séchés.

La coloration capillaire est évaluée de manière visuelle.

SIGNATURE

TRANSMIS A ET COMPRIS PAR (nom et signature)

DATE

NOMBRE DE LIGNES UTILISÉES

TEMOIN (nom et signature)

DATE



CAHIER N° 979

171

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Colorant support N1	colorant support C	pH	Oxydant	ype Cheveu	HT	Reffet
A	7+ 2948	Basique	H2O2 20V	BN	5	violine
				BP	4	violine puissant
A	808+2952	Basique	H2O2 20V	BN	6,5	Cendré
				BP	5,5	Cendré puissant
A	7+ 6	Basique	H2O2 20V	BN	5	Gris cendré puissant
				BP	3,5	Gris puissant
A	C7013+ 54192	Basique	H2O2 20V	BN	6	Gris cendré
				BP	5,5	Gris cendré violacé
A	C8181 + 66	Basique	H2O2 20V	BN	6	Gris bleu
				BP	5	Gris bleu
A	C7035+ 2484	Basique	H2O2 20V	BN	6	vert doré
				BP	5	

pH
neutre
6-11/2007
npx



SIGNATURE

M. Gendron

Suite page 172

NOMBRE DE LIGNES UTILISÉES

1 à 20

DATE

1.12.97

TRANSMIS A ET COMPRIS PAR (nom et signature)

Ade LABBEY

DATE

01/12/97

TEMOIN (nom et signature)

H. LAUNOUY

DATE

1.12.97